REQ.NO.1-9047

CONTRACT REPORT NO.OSU81-00186

Final report: Electron microprobe analysis of macerals in Canadian coals

Contract: 055U.23440-1-9047

CONTRACTOR:
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29pp

A program to study the influence of mineral matter on combustion using in-situ microprobe analysis commenced in October 1981. The research concentrated on 3 samples of coal from western Canada supplied by CANMET-ERL. These are:

- 1 lignite sample from Estevan, Saskatchewan (P-637);
- 1 coarse refuse bituminous coal sample from British Columbia (P-625); and
- 1 sub-bituminous coal sample from Forestbrook, Alberta (P-642).

The first stage of research dealt essentially with the preparation of samples and their pellets. The second stage constituted an in-situ microprobe analysis of these pellets in order to assess the influence of mineral matter on the combustion of these coals.

A) Procedures followed for preparation of samples and pellets

The determination of coal composition and chemistry requires the analysis of a representative split from a bulk sample. The entire sample, for each of the coals provided, was crushed to $\frac{1}{2}$ inch and a representative sample (150 grams) was then split out of it. Great care was taken to obtain sufficient representation of the various particle sizes present in the initial sample.

After labelling all samples with appropriate codes, the split out samples (150 grams) were ground by hand to -20 mesh.

A representative split of approximately 95 grams from the -20 mesh reserve was extracted and further ground to -60 mesh. Of this split, 75 grams was utilized for chemical analyses: proximate-ultimate analysis, ash content analysis, and BTU and FSI determination. The result of these analyses as well as a maceral point count for P-625 are included in Annex-1. The remaining 20 grams was used to obtain analysis of the ash content by plasma furnace combustion. This procedure was followed for each of the 3 coal

types under investigation.

Crushed particle pellets representative of each coal type were made for the petrologic analysis of coal composition and rank and for use in the second stage of the research. One pellet of each coal type was made, for a total of three.

In mounting the pellets, 15 grams of -20 mesh crushed coal was added in a previously cleaned steel mold (1 inch diameter by 1½ inch deep) to a polyvinylacetate epoxy resin mixture in the approximate proportion of 8 parts of coal to 5 parts resin. After the mold was labelled, its interior and the plugs were sprayed with a suitable releasing agent. After thoroughly stirring with a glass rod, the coal-epoxy mixture was placed into the mold, which had been fitted with a bottom plug. The mold was filled to the brim and the coal tamped down with a tamping rod. The mold was then sealed tightly, using a top plug with the help of a hydraulic press. The molds were then allowed to harden at room temperature for 24 hours. When the pellets were removed from the molds, they were ready for polishing.

For petrographic analysis an excellent scratch-free and relief-free polish is required. The procedure for polishing pellets used in the CANMET-ERL lab (and as outlined in the ASTM Annual Book, Part 26, 1979) was strictly followed and extreme care was taken to avoid contamination with stray grit.

Three additional pellets were mounted from the ash residues of each coal obtained from the plasma furnace combustion procedure. All pellets were then cut down to a thickness of 1 cm., in order to fit the mounting mechanism of the probe.

B) In-situ microprobe analysis of the coal pellets

Introduction

At the request of Dr. B. K. Nandi (CANMET), the second stage of the research was carried out at Dalhousie University, Halifax, on their Cambridge Mark-V Microprobe and Ortec Energy Dispersive System. The research was performed by Guy Masson, with the assistance of the staff at Dalhousie, from November 30 to December 5, 1981.

The object of this study of selected coal samples using an electron microprobe was to evaluate the feasibility of performing and to perform, if possible, the following tasks (i.e., from statement of work of CANMET Contract OSU81-00186):

1. Mineral distribution (Al, Si, Fe, Mg, Na, K, Ca, S, Ti, and P) shall be determined by in-situ microprobe analysis (Back-scattered or micrograph using K line) of: i)lignites; ii)sub-bituminous; and iii)bituminous coals provided by Energy Research Laboratories.
2. The analysis shall be repeated on vitrinite, exinite (reactive float), inertinite (sink) and fusinite components of the three samples and also on three combustion residue samples from Energy Research Laboratories. Micrographs shall be generated for all samples and the relationship between mineral content and coal constituents shall be established.

During the research, it became evident that these goals would be very difficult to achieve with this system.

Basic principles of operation of the electron microprobe

The basic operating principle of an electron microprobe is that an electron beam is focused onto a small area of a sample (polished mounts), from which X-rays characteristic of the chemical composition of the area are then emitted. The X-rays contain information on the energy, depending on the element emitting the X-ray photon, and intensity, which depends on concentration of the element. The emitted X-rays then are dispersed into their wavelength spectrum and the intensities of their spectral lines are measured quantitatively.

Two main modes of quantitative analysis are generally utilized. In the Wavelength Dispersive Spectrometers (W.D.S.), the X-rays emitted from a sample are dispersed by a suitable crystal standard (i.e., containing reference standards for the elements to be evaluated) arranged to satisfy the Bragg equation $n\lambda = 2d \sin\theta$. By varying θ, different values of λ are diffracted at different angles. Substitution of crystals with different d values extends the range of λ (Rucklidge, 1976). In the Energy Dispersive System (E.D.S.), the X-ray detector is a piece of highly purified Si, with a controlled amount of Li and the detector is held at liquid nitrogen temperatures. The electrons freed are swept out of the detector under the influence of an applied voltage, which produces a charge signal whose amplitude is a direct function of the X-ray photon energy (Rucklidge, 1976).

A detailed discussion of the various components' functions and the way they work, as well as a general understanding of electron microprobe instrumentation and systems can be found in Smith (1976) and Rucklidge (1976). A discussion of petrological applications of microbeam techniques is covered in detail by Clarke (1976).

Characteristics and limitations of the Cambridge Mark-V Microprobe

Quantitative and qualitative chemical analyses of inorganic material have been performed routinely at Dalhousie University with the Cambridge Mark-V Microprobe. The microprobe lab has a number of metal, synthetic oxide, sythetic sulphide and natural mineral standards that cover most of the common major elements (from sodium to uranium), as well as some trace elements including the rare earths.

Scanning capabilities can be performed by 1)spot in grains (in-situ) down to~ 5 microns in size (this is the usual method for

mineral analysis and can be qualitative or fully quantitative); 2)line scans, which plot composition vs. distance; and 3)area scanning for element mapping. This last method is important in demonstrating distribution and concentration of elements in minerals. The area scanned covers roughly the frame of the optical system of the probe (i.e., an area approximately $600\mu \times 350\mu$).

Both W.D.S. and E.D.S. quantitative analyses can be performed on the system. The characteristic results obtained and the limitations for both types of analysis are listed in Table.1.

Standards and methods used during investigation

The standards used during the investigation and their peak (angle) values are listed in Table 2. Mineral distribution was undertaken for S, K, Ca, Si, Al and Na for various types of macerals for the P-625, P-642 and the Sydney reference pellets. These elements were selected because they were the elements giving the best peak responses as observed on the Ortec 6200 Multichannel Analytical Oscillograph screen for numerous readings of the coals to be investigated. Unfortunately, carbon values could not be observed on the screen, since the system can only analyzed for elements from Na to U and also because no standard is available for C in this system.

At the time of work, mechanical probems were encountered with the standard for the element sulfur. This standard was off in the observed values by one order of magnitude.

The elements Fe, Ti and P were not assessed because of their location on a different crystal than the ones used for S, K, Ca, Si, Al and Na (see Table 2) and because the changing of crystals is a very time-consuming process. Cost and the limited amount of time available for research limited us to a study of the six

elements previously listed.

For the readings taken, the spot in grains method and the area scanning method were used.

Problems related to identification of macerals

Coal macerals normally observed by reflected light in an optical microscope (with oil-immersed lens) can also be identified in the emission images of back-scattered secondary electrons, provided that no coating is added to the surface of the polished mount--the coating of a polished coal pellet having as its main effect the obliteration of the differences in reflectance between the various types of macerals, which is the main means of maceral identification.

With the Cambridge Mark-V Microprobe, polished mounts (i.e., pellets) can be viewed only in reflected light in probe and all samples have to be carbon coated prior to use in probe in order to avoid deflection of the scanning beam by ionization of the mineral particles. Despite the fact that only a minimal amount of carbon coating (50-100Å) was applied to the pellets' surfaces, very few macerals could be identified with certainty following this coating of the pellets.

Differences in macerals' internal structures and comparison with pre-identified macerals from a pellet of bituminous coal from the Sydney Coal Field, Nova Scotia, were used as means to identify macerals from the western Canadian coals being analyzed. The low ash content≃(3%) and high vitrinite content of the Sydney coal pellet helped in the identification of some macerals from the high ash (38%) and high semi-fusinite content of the P-625 and P-642 western Canadian coals. By this method, several areas containing a variety of maceral types were identified. This also allowed us to explore the possibility of evaluating the distribution and

chemical character of the mineral matter in the maceral types by using the X-ray electron-probing capability of the system and by taking superimposed X-ray photographs for various elements (essentially S, K, Ca, Si, Al and Na) for a specific maceral or group of macerals from a selected area.

The study of the lignite (P-637) was abandoned because of the complex nature of the macerals to be identified.

C) Discussion of results obtained

Distribution of elements from mineral matter in macerals

Probe readings were taken from identified individual macerals using both the spot in grain and the area scanning methods on the W.D.S. system. The former was found to be particularly useful in obtaining a reading of only the pure maceral material, while the latter tended to average out the content of the maceral matter and the various inclusions encountered within the maceral scanned.

After the probe was focused on a maceral, a reading was taken for the six elements Si, K, Ca, Na, Al and S for a period of 200 seconds. The response curve obtained from the reading and shown on the Ortec System Oscillograph screen was then studied. The peaks were read and evaluated with the X-ray Critical-Absorption and Emission Energies Chart of the elements. Those were then recorded by decreasing intensity of the peaks. This was repeated for identified vitrinite, semi-fusinite and fusinite macerals from the Sydney, N.S., the P-625 and the P-642 pellets. Mineral matter grains from the ash pellets for the P-625 and P-642 coals were also probed. The results obtained are shown in Table 3. It was mechanically impossible to obtain photographs of the macerals probed or printouts of the response curves observed on the

Oscillograph screen.

Some interesting observations can be drawn from Table 3. For all the coals studied:

- Na and K have little significance;
- Ca is highest in fusinite and decreases toward vitrinite;
- Si occurs in greater concentrations in vitrinite than in semi-fusinite or fusinite;
- high S content and great variability of Al and to some extent Si are related to the presence of opaque inclusions (i.e., probably pyrite crystals and/or clay inclusions);

The spot in grain scanning method "cleans up" the results by eliminating the averaging-out effects obtained in the area scan approach. However, this method is preferable for the reading of a maceral that would cover most of the frame of the optical system of the probe.

The higher mineral matter content in the P-625 and P-642 coals made the selection and identification of acceptable macerals in those coals a very difficult and tedious job. In addition, because the identification of semi-fusinite under those conditions is very uncertain, no good intermediate results between vitrinite and fusinite could be obtained. In some cases Si is higher than Ca, but in others both are equal or even Ca is higher than Si.

In the absence of good reflectance contrast, only fusinite with good internal structures and very homogeneous vitrinite could be identified with some certainty. It was impossible to identify exinite or resinite.

Distribution of elements from mineral matter in selected areas

Areas the size of the frame of the optical system exhibiting a variety of maceral types were then selected and X-ray photographs of S, Si, Al, Ca, Na and K elements were taken for each, in order to outline trends of mineral matter occurrences by the

superimposition of all the photographs within each given area photographed.

One area was photographed for the Sydney coal pellet and two from the P-625 coal pellet. These are illustrated in Plates 1 to 3. Elements that only showed background values in the photograph (such as in Plate 3E, F) were not included in the plates. Perhaps a greater exposure time would have revealed points of concentration for these elements.

The following relationships between some of the elements photographed can be observed in Plates 1 to 3:

-Superimposition of Al and Si in some restricted areas (Plate 1A and C, Plate 3A and C) possibly indicates the presence of clay fragments and/or silica grains with clay inclusions;

-The Si content in Plate 2B shows good scattering through several vitrinite fragments, while Ca (Plate 2A) shows some degree of superimposition with Si and indicates a semi-fusinite to low fusinite composition;

-In Plate 3B, C, D, E the elements are almost at background level and no occurrences can be observed except for few restricted areas for Ca (Plate 3B).

Interpretation of the superimposition patterns observed for certain elements (i.e., mainly Si and Al) are quite unsatisfactory, mainly because of the absence of the outlining of the macerals and various grains covering the areas selected. Because of this the mineral matter occurrence and distribution cannot be related to specific maceral types with this method. It is therefore essential that the area to be probed be photographed (under the reflected light microscope) and each maceral and grain present identified before probing and X-ray photographing is attempted.

Quantitative analyses

Attempts to obtain quantitative results for selected macerals having good curve responses for the same basic 6 elements were unsuccessful both for the E.D.S. and W.D.S. systems.

The failure of the E.D.S. system was attributable to the absence of C standard as a major element in the program of the system. The minor elements present are thus read as background value since they cannot be linked to a major element and no percentage can be assigned to their values read by the probe. It might be possible to succeed with this system by feeding into the program a fixed value for C by the use of the FIXED ENTRY clause. Once C is entered in the program as a fixed weight value, the computer could correct and adjust for the other standards read for the minor elements present. This possibility will be tested by the staff of the Dalhousie microprobe lab and the results obtained should be communicated to the author in the near future.

Quantitative results could have been obtained by the W.D.S. system if an appropriate C standard had been available. However, in addition to the difficulty of establishing a suitable C standard, other disadvantages of this method would be cost and the limited amount of data obtainable in a day (i.e., 8-12 analyses under the best conditions per day, as opposed to 8 analyses per hour for the E.D.S. system). A great amount of time is also necessary to adapt the system to perform quantitative coal analysis.

D) Conclusion: recommendations

Despite the failure to fulfill the tasks outlined in the contract statement (see p. 3) by using the probing capabilities of the Cambridge Mark V Microprobe model, it is believed that all of these goals can be achieved using a more appropriate approach.

It is therefore recommended that the following steps be taken in order to insure completion of the work.

1) From a standard pellet (i.e. 2.5 cm in diam., 1.0 cm thick) select an area sharing the greatest diversity of maceral types. This area should be as close as possible to the size of the frame of the optical system (i.e. 600µ x 350µ);
2) Take a microphotograph of the area using the camera attachment on the reflected light microscope (i.e. lens under oil immersion) such as the one in use in the CANMET-ERL at Bell Corners;
3) Mark on the pellet the area photographed by the microfracturing method or by a system of coordinate marking or labelling on the sides of the pellet;
4) Identify on the microphotograph all macerals and mineral matter fragments. This can be done by a direct labelling with letter code on the microphotograph or on an overlay covering the microphotograph, on which all grains are traced and labelled;
5) If the microphotograph taken covers an area greater than the frame of the optical system, select and measure an area roughly 600µ x 350µ showing the greatest diversity of maceral types. This area should be outlined on the microphotograph;
6) Coat pellet with a carbon coating of a thickness of 50 to 100Å
7) The scanning capabilities should be performed by
   a) spot in grain - for selected spot ≃ 5µ in diameter;

b) area scan - if entire area is to be probed.

It is recommended, in order to obtain more realistic results, that many spot-in grain readings be taken for specific macerals or grains and then all values be averaged.

It is also recommended that a system similar to the Philips SEM 505 model be used rather than to try to make the necessary adjustment in programming and standards on the Cambridge Mark V Microprobe. The Philips system can perform combined quantitative analysis, using both energy- and wavelength-dispersive techniques (W.D.S. and E.D.S.).

Rapid, quantitative, simultaneous elemental analysis over the range from sodium to uranium can be obtained with the E.D.S. system. This system uses a cooled, lithium-drifted silicon detector protected by a thin beryllium window. A special unit with removable window, called ECON, is available for light-element detection down to carbon. The system provides optimum detection conditions for point analyses, elemental mapping and elemental line scans. With this combination, identification of major components is made very simple by automatic peak-identification facilities. Elemental distribution or relative distribution of elements and background can be mapped and related to other images of the same area (i.e. X-Ray photographs).

Fully automated quantitative and qualitative analysis of all elements down to beryllium (i.e. C included) is also available with the W.D.S. on this model. In addition, the model is fully automated and permits the taking of high-resolution SEM micrographs which would be extremely useful for the study of mineral matter distribution in coal maceral types.

References

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TABLE 1 COMPARATIVE TABLE OF RESULTS EXPECTED AND LIMITATIONS FOR W.D.S. AND E.D.S. SYSTEMS

W.D.S.	E.D.S.
High quality quantitative analysis (± 1% for major elements)	Good quality analysis (±2%)
12 to 20 analyses a day depending upon number of elements involved (for mineral analysis usual number 8-12)	Up to 60 analyses a day (typically 8 analyses per hour)
Detection limit for minor elements down to .01% (100ppm) (i.e. detection below 1000ppm depending on material studied)	Detection limit for minor elements down to ≃ .1% Na ≃ .3% and Mg ≃ .2%
Can analyse for elements from Na to U	Can analyse quantitatively for elements from Na to Zn
Raw data must be tabulated and submitted to CDC 6400 by card deck (i.e. corrected results usually available by next day)	Raw data automatically corrected. Results for each analysis usually available within 10 minutes.

DATA SOURCE FOR THIS TABLE - PERSONAL COMMUNICATION FROM STAFF OF MICROPROBE LAB., DALHOUSIE UNIVERSITY

TABLE 2 STANDARDS USED FOR ANALYSIS

CHANNEL	CRYSTAL	STANDARDS (ELEMENTS)	PEAK ANGLES
#1	PET	K	50°48'
		Ca	45°18'
		Ti	36°48'
		S	75°56'
		P	89°22'
#2	RAP	Na	54°12'
		Mg	44°21'
		Al	37°03'
		Si	31°24'

TABLE 3 ELEMENT DISTRIBUTION IN SELECTED MACERAL TYPES

oal Pellet	Maceral type identified	Description of spot or area probed	Scanning method used	Order of peaks observed on Cr Oscillograph
Sydney, N.S.	semi-fusinite	milky homog., w/ streaks and opaque spots	area scan	S>Si>Al, no peaks K, Ca, Na
	vitrinite	clean milky w/ little inclusions	area scan	S>Si>Al, no peaks K, Ca, Na
	fusinite	w/ opaque inclusions	area scan	S>Ca>Si>Al, no peaks K, Na
	vitrinite	homogeneous spot	spot in grain	Si>Al>S>Na>Ca, no peak K
	vitrinite	pure spot	spot in grain	Si≈Al≈S=>Na>Ca, no peak K
	fusinite	homogeneous spot	spot in grain	Ca> S>Al>Si≫Na, no peak K
P-625	semi-fusinite	w/ opaque inclusions	area scan	Ca=Si>Al>S, no peaks K, Na
	vitrinite	w/ opaque inclusions	area scan	S>Si>Al, no peaks K, Ca, Na
	vitrinite	homogeneous spot	spot in grain	Si>Al>S>Na>Ca, no peak K
	fusinite	w/ opaque inclusions	area scan	S>Ca>Si>Al, no peaks K, Na
	fusinite	homogeneous spot	spot in grain	Ca>S>Si=Al>>Na, no peak K
P-642	vitrinite	clean milky w/ no inclusions	area scan	Si>Al>S Na Ca, no peak K
	semi-fusinite	w/ ≈40% opaque inclusions	area scan	S>Si>Al>Ca, no peaks Na, K
	fusinite	homogeneous spot	spot in grain	Ca>S>Al>Si≫Na, no peak K
	semi-fusinite	homogeneous spot	spot in grain	Si>Al>Ca>S, no peaks Na, K

Ash content
P-625 and P-642 No problems in identification and chemical content encountered for example

Quartz usually showed the following pattern Si≫Al≫S

Clay usually showed the following pattern Al>Si≫S

in which S and remaining elements would vary greatly w/ inclusions present

Elements probed: S, Si, Al, Ca, Na, K

ANNEX 1

DATA INCLUDED CONCERNING COALS STUDIED

P - 625 B.C. BITUMINOUS COAL

PROXIMATE ANALYSIS

ULTIMATE ANALYSIS

CALORIFIC VALUE

POINT COUNT OF MACERALS (i.e. 1000)

P - 637 LIGNITE, ESTEVAN, SASK.

PROXIMATE ANALYSIS

ULTIMATE ANALYSIS

CALORIFIC VALUE

P - 642 SUB-BITUMINOUS, FORESTBROOK, ALB.

PROXIMNATE ANALYSIS

ULTIMATE ANALYSIS

CALORIFIC VALUE

COMPARATIVE PELLET FROM BITUMINOUS COAL, SYDNEY, N.S.

PELLET IS FROM HARBOUR SEAM FROM #26 COLLIERY, SYDNEY, N.S.

FROM MIDDLE OF THE SEAM

≈3% ASH CONTENT

65% C

0.5% S

SOLID FUEL ANALYSIS LABORATORY
REPORT OF ANALYSIS
HEAD: LOUIS C. G. JANKE

P-625 B.C. Coal

SAMPLE NUMBER 4224-81
I.D. NUMBER N-81135
DATE REC'D 22-10-81

****

	AS REC'D	DRY	
PROXIMATE ANALYSIS			
			d.a.f.
Moisture	0.47		
Ash	38.80	38.98	
Volatile Matter	15.93	16.01	26.2
Fixed Carbon (By Difference)	44.80	45.01	73.8
ULTIMATE ANALYSIS			
Carbon	52.20	52.45	86.0
Hydrogen	2.77	2.78	4.56
Sulphur	0.22	0.22	0.36
Nitrogen	0.50	0.50	0.82
Ash	38.80	38.98	
Oxygen (By Difference)	5.04	5.07	8.3
CALORIFIC VALUE			
Calories per gm.	5021	5045	8268
BTU per lb.	9038	9081	14882

REPORTED BY: _____ DATE: 16-11-81

FSI NA

P - 625 B.C. COAL Coarse Refuse - Marceral Point Count

	100	200	300	400	500	600	700	800	900	1000	%
Vitrinite	49	101	150	200	256	306	365	423	482	534	53.4
Exinite + Resinite	1	3	3	3	3	3	3	3	3	3	0.3
Micrinite	1	3	5	7	8	9	11	11	14	14	1.4
Semi-Fusinite	37	76	113	151	187	230	263	296	330	359	35.9
Fusinite	12	17	29	39	46	52	58	67	76	90	9.0

This is a bituminous coal

53.4% VITRINITE

0.3% EXINITE

1.4% MICRINITE

35.9% SEMI-FUSINITE

9.0% FUSINITE

This coal also has

38.98% ash

45% fixed carbon

0.22% sulphur

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REPORT OF ANALYSIS
HEAD: LOUIS C. G. JANKE

P-63 7 lignite

SAMPLE NUMBER 4222-81
I.D. NUMBER N-81133
DATE REC'D 22-10-81

****

	AS REC'D	DRY
PROXIMATE ANALYSIS		
Moisture	19.27	
Ash	9.31	11.53
Volatile Matter	38.43	47.60
Fixed Carbon (By Difference)	32.99	40.87
ULTIMATE ANALYSIS		
Carbon	46.79	57.96
Hydrogen	2.57	3.18
Sulphur	0.55	0.68
Nitrogen	0.85	1.05
Ash	9.31	11.53
Oxygen (By Difference)	20.66	25.60
CALORIFIC VALUE		
Calories per gm.	4181	5179
BTU per lb.	7526	9322

REPORTED BY: _____ DATE: 16-11-81

FSI NA

SOLID FUEL ANALYSIS LABORATORY
REPORT OF ANALYSIS
HEAD: LOUIS C. G. JANKE

D-642
Sub-bituminous

SAMPLE NUMBER 4223-81
I.D. NUMBER N-81134
DATE REC'D 22-10-81

****

	AS REC'D	DRY
PROXIMATE ANALYSIS		
Moisture	5.30	
Ash	33.83	35.72
Volatile Matter	28.35	29.94
Fixed Carbon (By Difference)	32.52	34.34
ULTIMATE ANALYSIS		
Carbon	43.57	46.01
Hydrogen	2.54	2.68
Sulphur	0.42	0.44
Nitrogen	0.78	0.82
Ash	33.83	35.72
Oxygen (By Difference)	13.56	14.33
CALORIFIC VALUE		
Calories per gm.	4001	4225
BTU per lb.	7202	7605

REPORTED BY: _____ DATE: 16-11-81

FSI NA

PLATE 1 X-RAY PHOTOGRAPHS OF ELEMENT DISTRIBUTIONS SELECTED AREA: SYDNEY COAL, N.S.

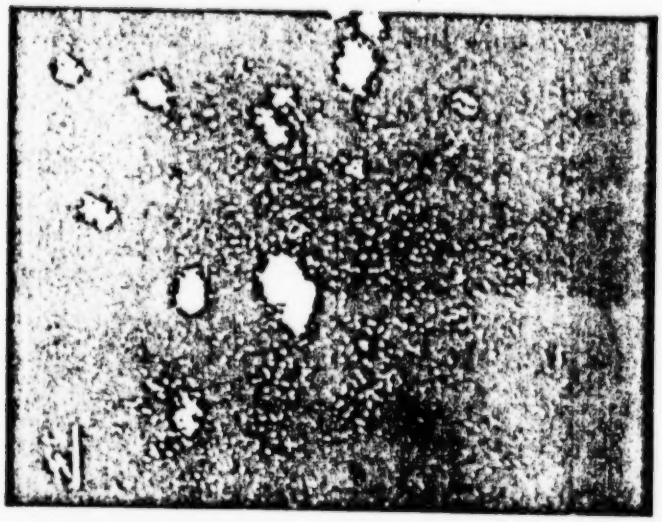
A Al 20 MINUTES EXPOSURE, 2ND HIGHEST PEAK

B Ca 20 MINUTES EXPOSURE, HIGHEST PEAK

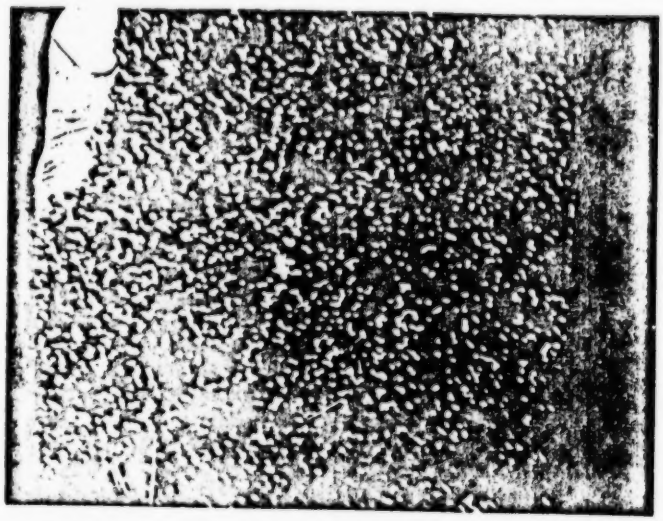
C Si 20 MINUTES EXPOSURES, 3RD PEAK

Plate 1

A



B



C

PLATE 2 X-RAY PHOTOGRAPHS OF ELEMENT DISTRIBUTIONS.

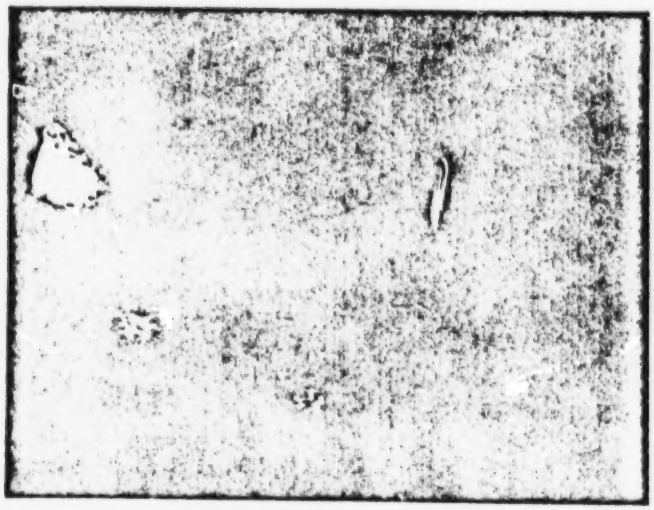
SELECTED AREA: BITUMINOUS, B.C. COAL (P -625).

A Ca 15 MINUTES EXPOSURE TIME, 2ND HIGHEST PEAK

B Si 15 MINUTES EXPOSURE TIME, HIGHEST PEAK

Plate 2

A



B

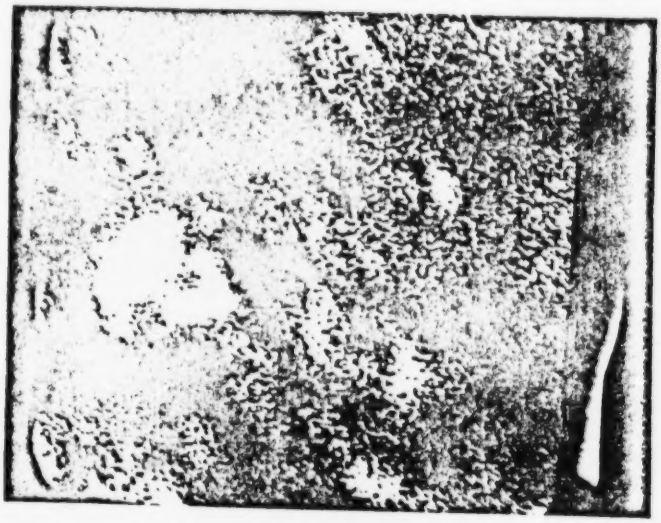


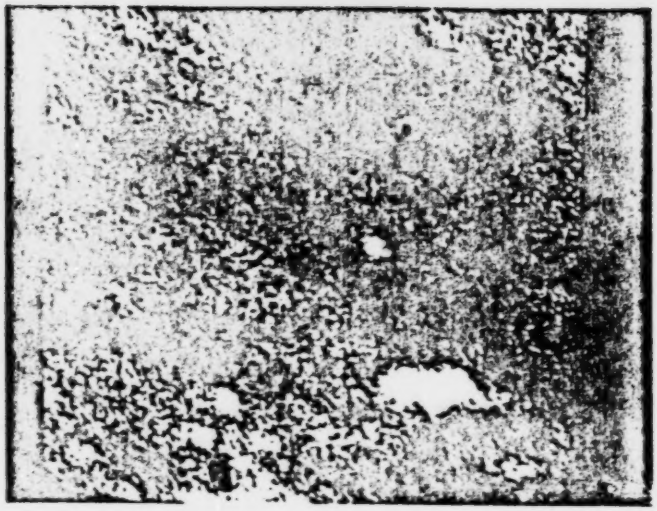
PLATE 3 X-RAY PHOTOGRAPHS OF ELEMENT DISTRIBUTIONS SELECTED AREA:

BITUMINOUS B.C. COAL (P – 625)

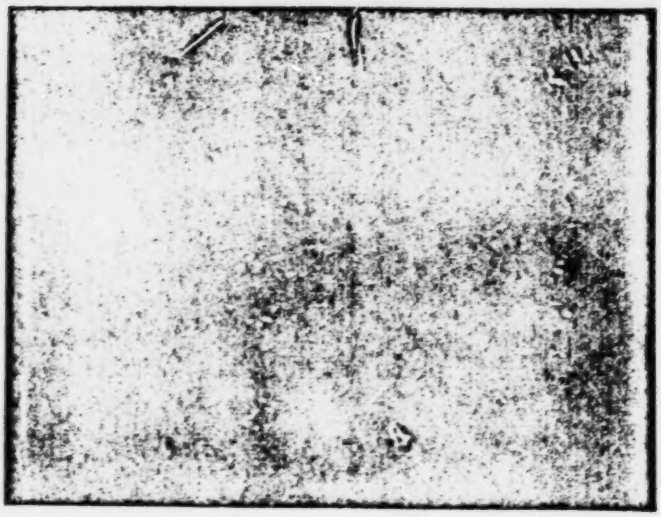
A	Al	15 MINUTES EXPOSURE
B	Ca	15 MINUTES EXPOSURE
C	Si	15 MINUTES EXPOSURE
D	K	15 MINUTES EXPOSURE
E	S	15 MINUTES EXPOSURE
F	Na	15 MINUTES EXPOSURE

Plate 9

A



B



C

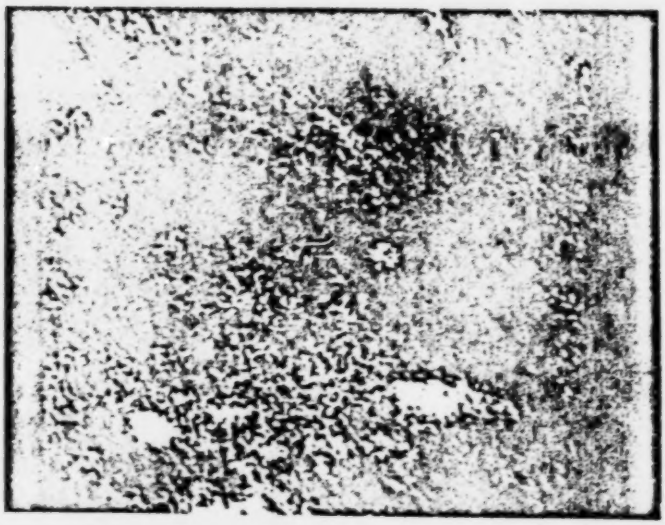
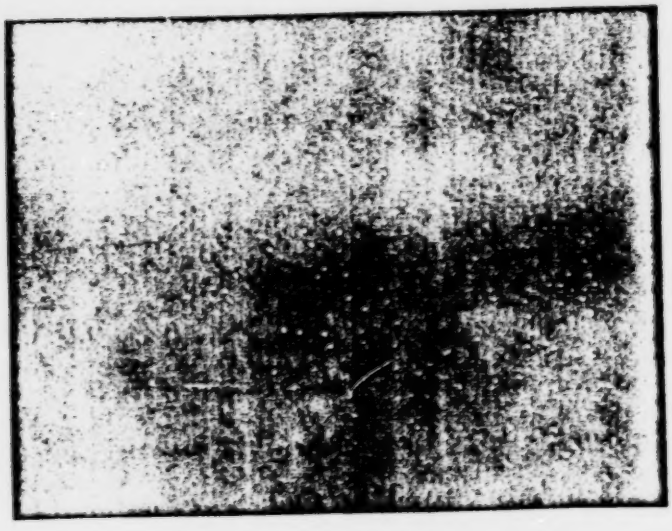


Plate 3 (cont.)

D

E



F